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Crystal structure of Cu(II)-bis-morpholine biguanide

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Copper (II)-bis-morpholine biguanide (Poddar 1968) crystallizes as deep red plates. Rotation and Weissenberg photograph show that the crystal belongs to the monoclinic system with cell dimensions

$$a=8.68\text{Å},\ b=12.74\text{Å},\ c=7.75\text{Å},\ \text{and}\ \beta=93.5°.$$

The only systematic absences were for $0k0$ with k odd and for $h0l$ with l odd, indicating that the space group is $P2_{1/c}$. Density determination by flotation method showed that there are two formula units of $Cu(C_6N_5OH_{15})_2$ per unit cell.

Three dimensional data were collected using multiple-film equi-inclination Weissenberg techique and CuK_α radiation. The intensities were estimated visually and corrected for Lorentz polarization and spot-size effects. The data were then brought to the absolute scale.

A three-dimensional Patterson synthesis showed that the copper atoms are placed at (0.0.0) and $(0.\frac{1}{2},\frac{1}{2})$ and the two ligands attached to copper have a trans- configuration. Due to their special positions, the copper contribute to the structure factors of $(k+1)$ even group of reflections only. Therefore, the three-dimensional Fourier synthesis calculated with the signs of the copper atoms showed a pseudosymmetry. The spurious mirrors at $y=0$ and $y=\frac{1}{2}$ made the correct identification of the lighter atoms very difficult, though it became evident that the four nitrogens around copper had a square planar configuration.

To avoid this difficulty symbolic addition procedure (Karle & Karle 1963, 1964 ; Karle *et al* 1964) of direct sign determination was applied with some modifications. For the $(k+1)$ even refletions the contribution of the copper atoms were subtracted from the F_0's, assuming that their signs are the same as those due to the copper atoms only. The new F's are thus due only to the lighter atoms of the structure. To these structure factors the Σ_2 formula

$$SE_{hkl} \simeq \sum_{k'l'} SE_{k'k'l'}\, SE_{h-k'k-k'l-l'} \qquad \text{...(1)}$$

was applied, where S means 'sign of'. The E values for all the reflections were calculated with the expression

$$E^2_{hkl} = |F_{hkl}|^2 / \epsilon \sum_1^N f_i^2 \qquad \text{...(2)}$$

where f_i is the scattering factor of the ith atom, ϵ is an integer, which in the case of group $P2_{1/c}$ is 2 for $h0l$ and $0k0$ reflections and 1 for all others. The origin was fixed by assigning positive sign to three reflections (210,021 and $\bar{2}1$) with very large E values. Starting from these initial signs the signs of many other reflections were generated by using equation (1). The probability of the sign of a particular E_{hkl} being positive was calculated with the formula,

$$P_+\left(E_{hkl}\right) = \frac{1}{2} + \frac{1}{2}\tanh\left(\frac{1}{\sqrt{N}} \mid E_{hkl} \mid \sum_{hkl} E_{h'k'l'}\, E_{h-h'k-k'l-l'}\right) \quad (3)$$

where N is the total number of atoms in the unit cell.

The $(k+1)$ odd group of reflections to which signs could be assigned by the above method have now been added in the next three-dimensional Fourier synthesis. This synthesis clearly shows that the spurious mirror is broken partially, so that the location of the light atom peaks is no longer a formidable task. The refinement of the structure is now in progress.

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